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NAVWEPS REPORT 7918

Part 1

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THERMAL STUDIES OF REINFORCED-PLASTIC MATERIALS

Part 1. DIFFUSIVITY OF FIVE REINFORCED-PLASTIC HEAT BARRIERS

by

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ABSTRACT. This report discusses a study conducted at the Naval Ordnance Test Station (NOTS) on the thermal properties of reinforced-plastic laminates at high temperatures. Thermal diffusivity data are given for five different laminates, and the ablation rate in mils per second is given for 15 different laminates.

A part of these studies was conducted at the University of New Mexico and will be reported as Part 2 (Properties of Nine Reinforced-Plastic Laminates) of this report.

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U. S. NAVAL ORDNANCE TEST STATION

China Lake, California

February 1963

U. S. NAVAL ORDNANCE TEST STATION

AN ACTIVITY OF THE BUREAU OF NAVAL WEAPONS

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FOREWORD

The information contained in this report represents the findings of an applied research study of the thermal properties of reinforced-plastic laminates at high temperatures. This study was conducted from February 1960 to March 1961 and was supported by Bureau of Naval Weapons Task Assignment RMMP-21-001/216-1/F009-01-016.

This report presents data on studies conducted at NOTS, and Part 2 (Properties of Nine Reinforced-Plastic Laminates), to be issued subsequently, will discuss studies conducted at the University of New Mexico under the direction of NOTS.

This report was reviewed for technical accuracy by W. K. Smith and R. J. Landry.

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INTRODUCTION

At the present time, there is no accurate method for determining the thermal properties of reinforced-plastic, heat-barrier materials at temperatures above that at which thermal decomposition occurs (approximately 500°F). Steady-state heat-transfer equations are inaccurate under conditions of pyrolytic decomposition, as the degradation process produces continuous changes in the thermal conductivity, specific heat, and density of the specimen. The evolution of gases produced by thermal decomposition of the material further complicates boundary layer conditions. Several studies of the thermal degradation of plastics (Ref. 1), steady-state ablation (Ref. 2), and thermal properties (Ref. 3) have contributed materially to an understanding of the problems involved.

The requirements for heat barriers in rocket motors vary, depending upon operating conditions such as burning time, mass gas flow, chamber pressure, combustion temperature of the propellant, gas flow pattern at ablation area, combustion products, and other environmental operating conditions. It is difficult to predict with accuracy what material will perform best under a given set of conditions without undergoing a comprehensive evaluation program.

A basic knowledge of the pyrolytic ablation mechanism is essential to the study of materials and their evaluation as temperature- and ablation-resistant heat barriers. Because of the inaccuracy and difficulty of obtaining specific thermal data at ablative temperatures, it was decided to use thermal diffusivity (α) as the measurement most representative of the thermal properties involved.

The values obtained from these tests may be used as guidelines by the rocket designers in solving heat-transfer problems related to the use of plastic laminates as heat barriers in rocket motors. As additional data are obtained, the degree of accuracy and reliability will be improved.

EXPERIMENTAL PROCEDURE

RADIANT HEATING TESTS

Reinforced-plastic heat-barrier laminates 4.5 by 4.5 by 0.180 inches were prepared using 100 lb/in² laminating pressure at a curing temperature of 375°F for 1 hour. The laminates were then postcured for 12 hours in an oven at 375°F. Resin content of all samples was within the range of 47 to 53%.

Chromel-alumel thermocouples were attached to each side of the specimen panel by bonding them with the same resin used in the laminate. Two of the panels were then bonded together under heat and pressure to give a sandwich structure with four thermocouples, one on each outer face and one on each inner face (Fig. 1 and 2).

A quartz-tube radiant heating panel was positioned on each side of the sandwich (Fig. 3 and 4). An electronically controlled programmed heat rise of approximately 5.5°F/sec was maintained within a temperature range of 200 to 1500°F.

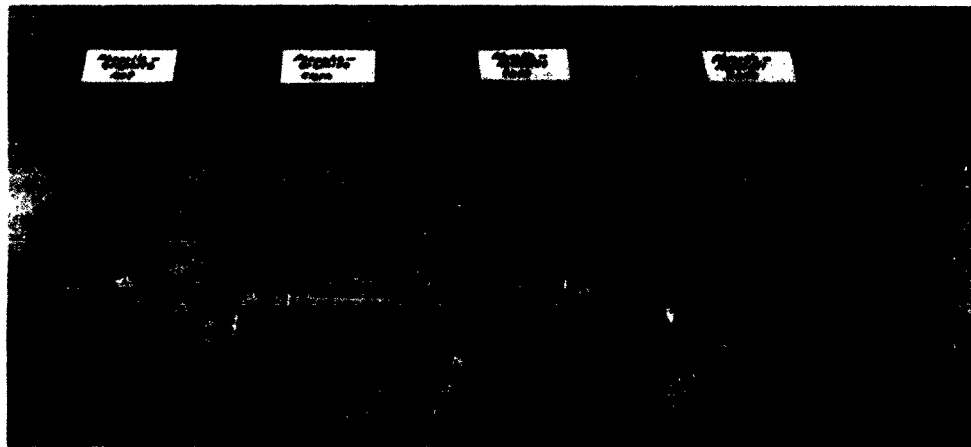


FIG. 1. Side of Laminated Test Specimen Before and After Heating.

The following types of reinforced-plastic heat-barrier laminates were evaluated for "apparent" thermal diffusivity:

Reinforcement (unpressed prepreg)	Thickness, in.	Resin
Asbestos cloth	0.125	Ironsides No. 101 phenolic
Asbestos cloth0625	Ironsides No. 101 phenolic
Refrasil015	91LD phenolic
Graphite cloth012	Ironsides No. 101 phenolic
Graphite mat5	91LD phenolic
Asbestos paper	0.010	Dow No. 2106 silicone

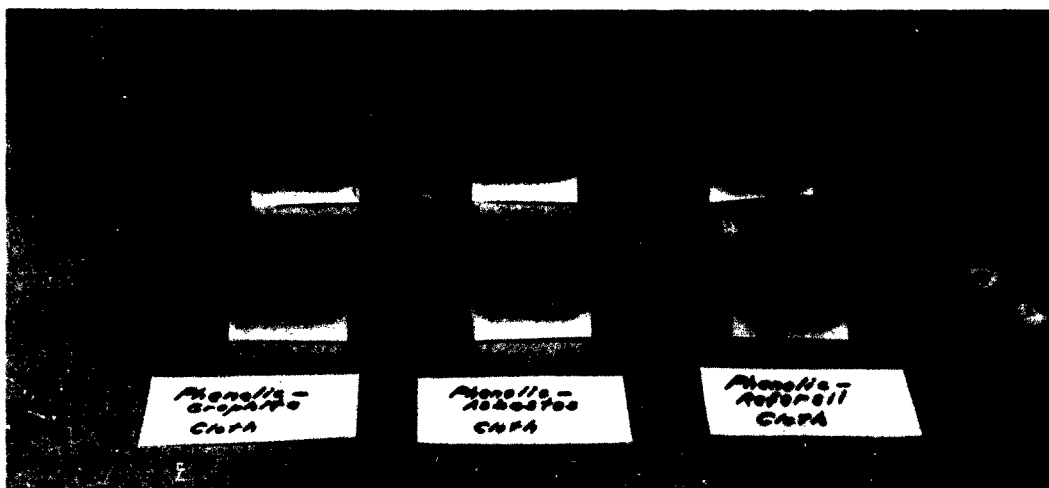


FIG. 2. Edge of Laminated Test Specimen After Heating.



FIG. 3. Thermal Diffusivity Equipment Using Quartz-Tube Radiant Heating.



FIG. 4. Quartz-Tube Radiant Heaters and Specimen Mounting.

The apparent thermal diffusivity was calculated at various temperatures from the experimental time-temperature curves for each material (Fig. 5-10). Thermal diffusivity was also plotted as a function of time for each panel. Duplicate panels were run in each test, and reproducible heating curves were obtained between the two panels and also between the same types of material run at different times.

Mathematical Method

Thermal diffusivity (α) is a function of thermal conductivity (K), specific heat (C), and density (ρ), and it is defined as

$$\alpha = \frac{K}{\rho C}$$

Using the experimental method developed by Butler and Inn, α was determined by the following mathematical method (Ref. 4 and 5):

$$\frac{\text{slope}}{T_1 - T_2} = \frac{2\alpha}{x_1^2 - x_2^2}$$

$$\alpha = \frac{(\text{slope})(x_1^2 - x_2^2)}{(T_1 - T_2) 2}$$

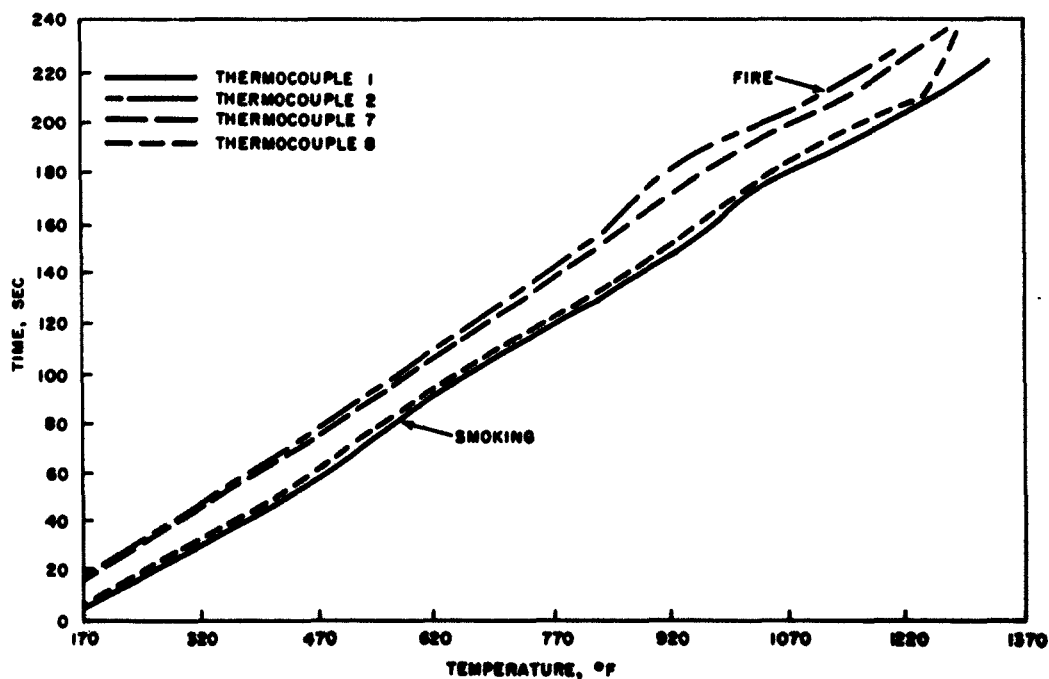


FIG. 5. Thermal Properties Test of Phenolic-Graphite Cloth.

where

$$\text{slope} = \frac{dT}{ds} \text{ (using parallel sections of the time-temperature curve)}$$

T = temperature, °F

S = time, sec

T_1 = outside thermocouple temperature, °F

T_2 = inside thermocouple temperature, °F

x_1 = distance of inner thermocouple from outer face of panel, in.

x_2 = distance of outer thermocouple from outer face of panel, in.

This experimental technique was based on a source of radiant energy of uniform distribution over the local plane. The intensity of the radiant energy could be programmed to give a linear temperature rise on the heated specimen surface with the specimen approximating an infinite slab insulated on the unheated surface. This determined the value of α , and its variation with temperature was also indicated.

Analysis

Phenolic-Graphite Cloth Panels. A slight thermal decomposition with some gassing is indicated at about 500°F in the phenolic-graphite cloth (Fig. 5), although it is not so pronounced as in some of the other materials. A major thermal phase change was noted at about 950°F. When the temperature rose above 1400°F, the sample became incandescent and all four thermocouples registered approximately the same temperature. Thermocouples 1 and 2 were on the inside and thermocouples 7 and 8 were on the outside. The two sandwiched panels produced very similar curves when heated simultaneously. The pyrolytic degradation phase changes were more pronounced in the time-temperature curves than in the thermal diffusivity-temperature curves, because of the scale used.

Phenolic-Asbestos Cloth Panels. Phenolic-asbestos cloth laminate was used as a heat-barrier standard, and although panels were made using both 0.065- and 0.125-inch-thick asbestos cloth, there was no appreciable difference in thermal properties between the two thicknesses. Thermal diffusivity ranged from 1×10^{-3} to 4×10^{-4} in²/sec, decreasing with temperature increases up to 1300°F (Fig. 6). The specimens showed some surface spalling and heat distortion, with a major thermal decomposition change noted at 550°F. Considerable charred resin remained in the asbestos matrix. Nonuniformity of composition was indicated in some cases by the difference in α between samples.

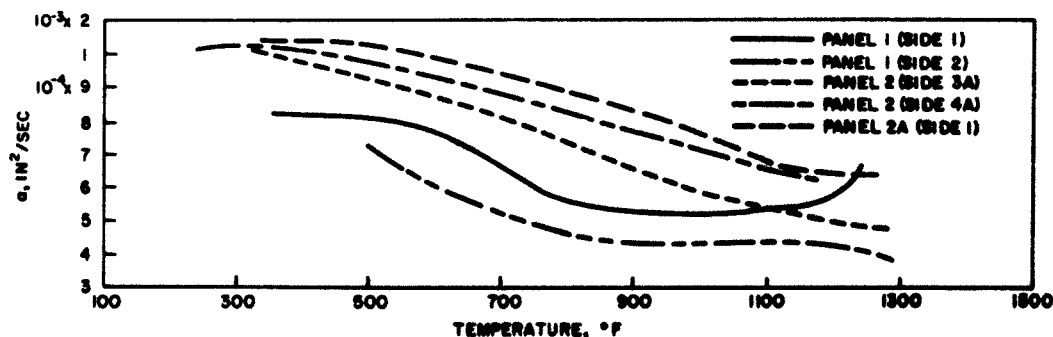


FIG. 6. Apparent Thermal Diffusivity of Phenolic-Asbestos Cloth.

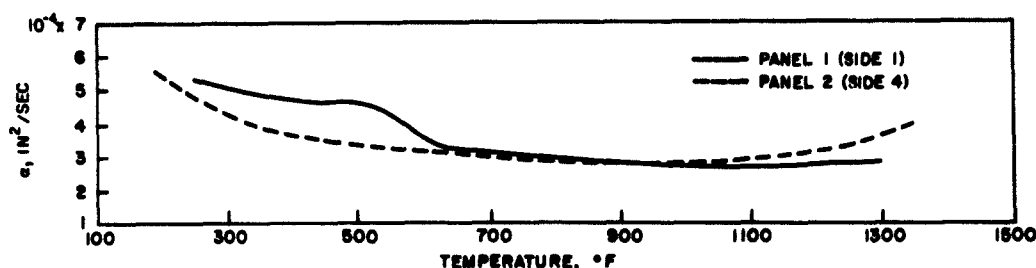


FIG. 7. Apparent Thermal Diffusivity of Phenolic-Refrasil Cloth.

Phenolic-Refrasil Cloth Panels. The thermal diffusivity-temperature curves for phenolic-refrasil cloth indicated excellent uniformity with only slight changes in α (6×10^{-4} to 3×10^{-4} in²/sec) over the temperature range (Fig. 7). Resin charring occurred at 550°F, as indicated by a change in the slope of the curve. The specimens showed some slight heat distortion and spalling.

Phenolic-Graphite Cloth Panels. The phenolic-graphite cloth and mat laminates were of considerable interest in this study because they had proved to be far superior in static-firing tests to any other laminates, particularly in a highly ablative environment. Ablation resistance was only partially indicated because in the radiant-heating tests there was no ablation caused by gas flow. Therefore, other tests such as static firings or plasma-jet tests, where ablation from heat and from high velocity gases are encountered, are required to establish ablation resistance.

As can be seen in Fig. 8, a definite thermal phase change occurred from 800 to 1000°F and not at 550°F as in the other materials. To better illustrate the thermal decomposition, actual experimental points were plotted on the curve of panel 3, sides 5 and 6, instead of drawing a smooth curve as in the other figures.

A microscopic examination of the panel nearest the radiant energy showed charred phenolic resin in the graphite cloth, although it appeared to be less than that of the other materials. There was no distortion of the panel and no visual damage of the graphite cloth. This material withstood the high temperature environment better than any of the other materials tested.

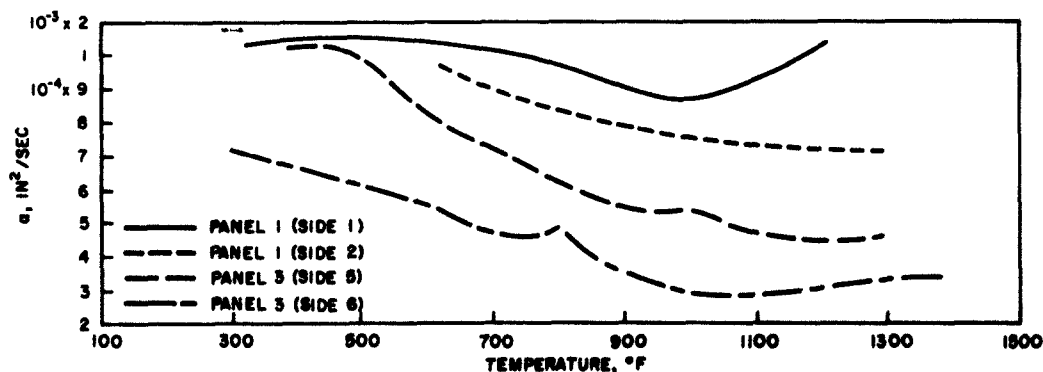


FIG. 8. Apparent Thermal Diffusivity of Phenolic-Graphite Cloth.

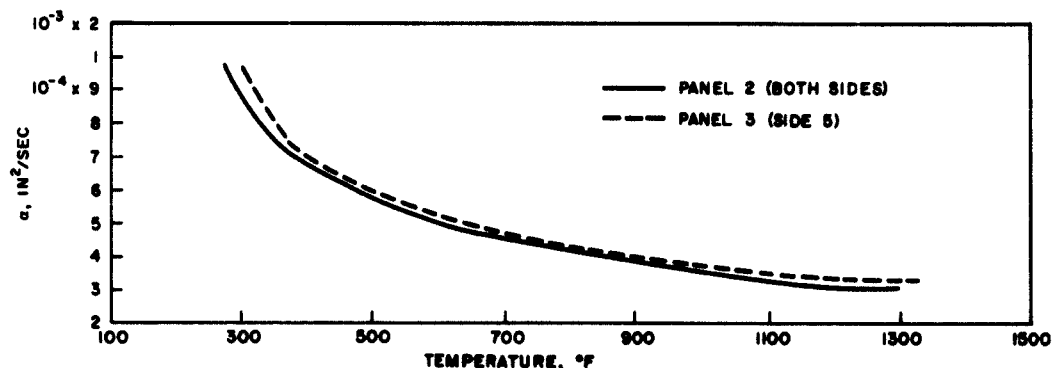


FIG. 9. Apparent Thermal Diffusivity of Phenolic-Graphite Mat.

Phenolic-Graphite Mat Panels. The phenolic-graphite mat specimens produced even, reproducible curves with no pronounced thermal phase changes (Fig. 9). Because of the structure of this material, its porosity was greater and its density less than the phenolic-graphite cloth. The phenolic resin charred in a uniform pattern, and the thermal diffusivity showed comparatively little change with temperature increases. The thermocouples broke on side 6 of panel 3, and these data were rejected. The sandwich was slightly warped but otherwise showed no visual damage.

Silicone-Asbestos Panels. The silicone-asbestos material gave consistent time-temperature curves, but because of delamination the thermal diffusivity was lower than for any of the other materials (Fig. 10). This indicates that the silicone-asbestos panel would perform well as a heat shield, but that it would be unsatisfactory under ablative environment because the loose sheets would be torn away very rapidly.

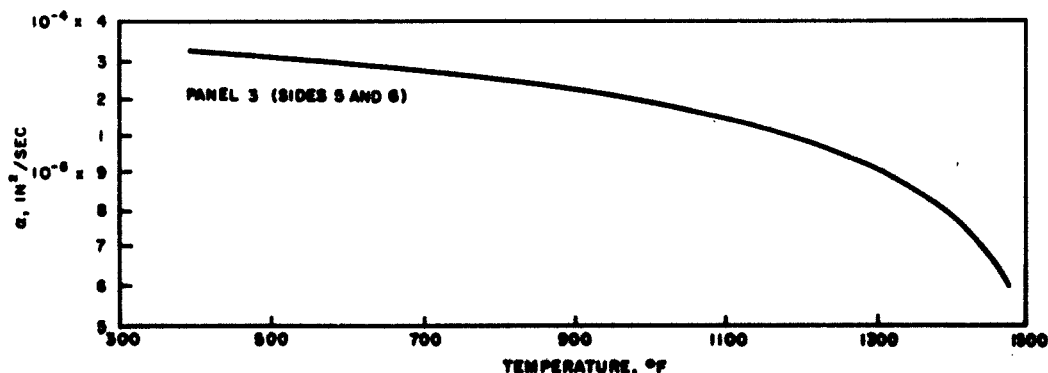


FIG. 10. Apparent Thermal Diffusivity of Silicone-Asbestos.

TABLE 1. THERMAL DIFFUSIVITY OF VARIOUS MATERIALS AT TEMPERATURES FROM 300 TO 1300°F

All data are for c (1×10^{-4}), calculated in square inches per second. Where the thermal diffusivity-temperature curves of all panels were similar, an average a value is shown.

Material	Temperature, °F														
	300			500			700			900			1100		
	Max.	Min.	Av.	Max.	Min.	Av.	Max.	Min.	Av.	Max.	Min.	Av.	Max.	Min.	Av.
Phenolic-asbestos cloth ^a	12.0	8.1	11.0	7.4	9.3	5.0	8.3	4.6	7.2	4.4
Phenolic-refrasil cloth ^b	5.0	4.3	3.5	3.1	2.8	2.9	2.6
Phenolic-graphite cloth	1.3 ^c	7.1 ^d	1.0	6.0	7.2	4.8	5.3	3.4	4.7	2.8
Phenolic-graphite mat ^e	9.7	6.4	4.8	3.8	3.2
Silicone-asbestos paper ^f	3.1	2.8	2.5	2.0	0.9

^a Five panels. ^b Two panels. ^c Thick panel. ^d Thin panel. ^e Average of three panels. ^f Average of two panels.

Results

An effort was made to compare the α values obtained from the radiant-heating tests of this study to those obtained by other investigators (Tables 1 and 2). Two sources of information on these values for various plastic laminates were found (Ref. 3 and 6), although the exact laminate compositions were not known except for one sample tested at NOTS (Ref. 6). Thermal diffusivity data were fitted to theoretical mathematical models as a check on the validity of the models (Appendix).

TABLE 2. COMPARISON OF APPARENT THERMAL DIFFUSIVITY VALUES

Material	Temperature, °F	$\alpha (1 \times 10^{-4})$, in ² /sec
NOTS Known Sample (Ref. 6)		
Phenolic-asbestos cloth	205	1.70
	500	1.59
	650	1.66
NOL (Ref. 3)		
Phenolic-glass cloth	392	63.0
	1022	51.0
	1472	95.0
NOTS Radiant-Heating Tests		
Phenolic-asbestos cloth	300	12.0
	1300	3.9
Phenolic-refrasil cloth	300	5.0
	1300	3.1

PLASMA-JET ABLATION TESTS

Tests were conducted at NOTS to determine thermal properties of various plastic laminates at high temperature (6000°F) and intermediate temperature (1400°F). Tests conducted at the University of New Mexico (NAVWEPS Report 7918, Part 2) were concerned with low temperature (300°F).

Thermal diffusivities of the laminates could be determined with a fair degree of confidence at the low and intermediate temperature ranges where a controlled temperature rise was maintained; but the high-temperature tests produced a rapid ablation rate with a corresponding non-uniform heat rise that invalidated the data for calculation of thermal diffusivity. The ablation rate in mils per second was determined for 15 different heat-barrier materials (Table 3) with a plasma-jet flame as the heat source. Test specimens were prepared in the form of laminates 4.5 by 4.5 inches and usually about 180 mils thick; however, in some cases they were as thick as 970 mils. Thermocouples were molded in the center of the specimen and on the side opposite flame impingement. Several proprietary specimens were received in various sizes that were un-

TABLE 3. ABLATION RATES OF HEAT-BARRIER MATERIALS

The flame temperature was 6170°F.

Material	Sample thickness, mils	No. samples tested	Ablation rate, mils/sec
With Thermocouples			
Phenolic-graphite cloth	185	4	12.0
Phenolic-graphite mat	360	1	10.3
Phenolic-refrasil cloth (high silica)	180	2	15.7
Phenolic-asbestos cloth plus titanium foil in center of laminate	195	1	19.7
Phenolic-asbestos cloth plus aluminum foil in center of laminate	192	1	20.9
Phenolic-asbestos cloth plus silver foil in center of laminate	185	1	14.7
G.E. silicone rubber No. SE 565	180	1	59.3
Without Thermocouples			
Johns-Manville GS 2048 insulation	367	1	114.7
Rocketdyne R-124 rubber....	187	1	69.3
Johns-Manville Min K 2000 insulation	920	1	108.3
Stoner rubber SMR-7	287	1	46.3
Cordo, phenolic-glass cloth	157	1	18.7
Rescot, phenolic-refrasil cloth	127	1	17.7
Phenolic-asbestos cloth....	177	3	22.9
Phenolic-refrasil cloth	175	1	24.9
Phenolic-asbestos cloth....	385	3	13.6

suitable for thermocouple embedment, and they were tested using a manual, visual control. When thermocouples were used, an oscillograph time-temperature record was obtained, and burn-through was determined the instant the thermocouple was destroyed by the ablating flame.

The temperature of the flame at the location of the sample was determined by inserting a 1/8-inch-diameter tungsten rod in the center of the flame and longitudinal to the plasma jet. The rod was allowed to melt along its axis until equilibrium occurred, and then the distance from the

end of the rod to the plasma arc head was measured. This method of determining the flame temperature was checked several times during the ablation tests and gave good reproducibility. The melting point of tungsten (6170°F) was established as the ablation temperature, and this point (1 inch from the arc face) was the distance to the inside face of the sample at the start of the test. A specimen holder, activated by a hydraulic cylinder, positioned the test specimen at this distance in the flame and was capable of swinging the specimen in and out of the flame as needed.

Graphite cloth and graphite mat impregnated with phenolic resin showed superior ablation resistance compared to the other materials (Table 4). A considerable difference in ablation rate was determined by the thickness of the sample. The thicker the sample, the less the ablation rate became for the same material. (This could have been caused by the increased over-all flame distance at the end of burning.) Comparison of the various materials can only be considered valid, therefore, for those specimens that were of approximately the same thickness.

TABLE 4. COMPARISON OF PLASTIC LAMINATES

Material	Ablation resistance	Thermal diffusivity	Resistance to heat damage	Numerical rating ^a
Phenolic-graphite cloth	excellent	medium	excellent	1.0
Phenolic-graphite mat	good	low	good	1.5
Phenolic-refrasil cloth	good	low	fair	2.0
Phenolic-asbestos cloth	fair	medium	fair	2.0
Silicone-asbestos paper	poor	very low	poor	3.0

^a Arbitrary numerical rating based on heat-barrier effectiveness in actual static-firing rocket-motor tests, radiant-heating tests, and plasma-jet heating tests. Best material is rated 1.0.

COMPARATIVE RATING OF PLASTIC LAMINATES

Based on the data obtained from static-firing,¹ radiant-heating, and plasma-jet-ablation tests, the various plastic laminates were given a comparative rating (Table 4).

CONCLUSIONS AND RECOMMENDATIONS

The determination of thermal diffusivity does not in itself offer sufficient data for the selection of a heat-barrier material. However, in combination with qualitative factors such as pyrolytic effects on structure, fiber failure, degradation of resin, and ablation resistance, it is possible to make a more intelligent selection of materials than can be made from static-firing tests alone.

The radiant-heating equipment and electronic controls (Fig. 3 and 4) were established on a temporary basis and could be improved by a more permanent arrangement. Because the programmed heat rise fluctuated slightly, a closer control and a faster heating rate are desirable.

¹ U. S. Naval Ordnance Test Station. Research and Development of High Temperature Heat Barrier Materials, by W. E. Donaldson, U. L. Johns, and H. G. Chase. China Lake, Calif., NOTS, 1960. (HDP 1087.)

Appendix

FIT OF TEMPERATURE CURVES
TO MATHEMATICAL MODEL²

Data taken in tests of liner materials to withstand heating have been fitted with curves based on a mathematical model of the heat transfer (Tables 5 and 6 and Fig. 11 and 12). The tests were carried out on samples prepared by binding together two panels of the test material in a sandwich with the temperature rise readings taken from the thermocouples in the middle. Heat was applied from large high-temperature radiating sources on both sides of the sandwich panels at an essentially constant rate.

The mathematical model was developed in detail in Ref. 7. Briefly, the theoretical model uses a transfer coefficient for the surface to which heat is applied, a similar, independent coefficient for heat transfer from the "cold" face, and a conductivity coefficient for heat transfer within the material. These were denoted, respectively, by a_i , a_o , and a_1 both in this report and in Ref. 8, except that in this report a_1 is defined by $\tau = a_1 t$ or, equivalently, $a_1 = 1/kL^2$. The model assumes that, initially, the material is uniformly at the temperature of the cold side and that after heat is applied the temperatures on both sides remain constant for the duration of the experiment.

This model was adapted to the work described in this report by treating each half of the sandwich as an independent test. Using $a_o = 0$ takes into consideration that placing the two halves of the sandwich back to back prevents heat loss from the cold side. The model is an approximation because the following assumptions were made: (1) instantaneous temperature rise at $t > 0$, while there was some time lapse in the actual experiment; (2) a known temperature on the hot side, where it was considered most appropriate to use the terminal temperature; and (3) a_i , a_o , and a_1 were constant.

²U. S. Naval Ordnance Test Station Memorandum, 5077/RSC of 1 Nov. 1960, to W. E. Donaldson, Subj: Curve Fits to Temperature Data.

TABLE 5. CURVE FITS OF THEORETICAL AND
EXPERIMENTAL TEMPERATURE DATA FOR
SILICONE ASBESTOS

Time, sec	Temperature, °F	
	Observed	Fit
Thermocouple 2: $\alpha_1 = 0.24$, $\alpha_1 = 0.025$, RMS = 16.6		
0	100	100.00
61	463	437.87
91	589	584.89
121	713	709.45
152	815	818.19
182	896	907.10
211	970	980.10
240	1034	1042.29
267	1090	1091.89
284	1140	1119.52
320	1178	1170.13
348	1219	1203.09
375	1257	1230.39
404	1285	1255.51
432	1310	1276.23
460	1318	1293.98
488	1317	1309.18
516	1319	1322.20
544	1325	1333.36
572	1338	1342.91
600	1353	1351.10
627	1370	1357.88
655	1383	1363.92
685	1392	1369.43
714	1397	1373.96
744	1398	1377.94
773	1398	1381.21
Thermocouple 7: $\alpha_1 = 0.24$, $\alpha_1 = 0.025$, RMS = 35.3		
0	100	100.00
43	446	342.70
73	506	508.84
113	591	691.57
132	695	765.19
163	815	869.88
193	914	955.47
221	1000	1023.51
249	1087	1081.80
277	1140	1131.73
304	1187	1173.08
331	1226	1208.70
358	1260	1239.38
387	1293	1267.61
415	1316	1290.90
443	1326	1310.84
471	1329	1327.93
499	1337	1342.57
527	1348	1355.10
555	1364	1365.84
581	1374	1374.43
610	1388	1382.66
638	1400	1389.45
667	1412	1395.46
696	1418	1400.57
727	1420	1405.21
755	1425	1408.76
781	1423	1411.60

TABLE 6. CURVE FITS OF THEORETICAL AND
EXPERIMENTAL TEMPERATURE DATA FOR
PHENOLIC-GRAPHITE MAT

Time, sec	Temperature, °F	
	Observed	Fit
Thermocouple 2; $\alpha_1 = 3.32$, $\alpha_1 = 0.0043$, RMS = 54.6		
53	442	362.81
83	650	634.94
111	825	855.66
140	972	1047.48
169	1110	1206.83
197	1242	1334.89
225	1377	1441.79
253	1500	1531.03
281	1600	1605.54
309	1689	1667.73
338	1766	1721.33
366	1836	1764.40
395	1867	1801.51
425	1896	1833.26
454	1915	1858.63
484	1925	1880.33
514	1935	1898.22
544	1937	1912.95
574	1940	1925.10
605	1945	1935.41
Thermocouple 7; $\alpha_1 = 3.32$, $\alpha_1 = 0.0043$, RMS = 55.7		
36	300	210.05
66	552	483.93
95	752	733.99
124	917	946.02
152	1064	1117.04
180	1196	1259.91
208	1315	1379.20
236	1437	1478.78
264	1532	1561.92
292	1644	1631.32
321	1732	1691.13
349	1810	1739.18
378	1861	1780.60
407	1888	1814.95
436	1915	1843.45
466	1934	1867.82
496	1940	1887.90
525	1945	1903.95
555	1952	1917.68
585	1958	1928.99

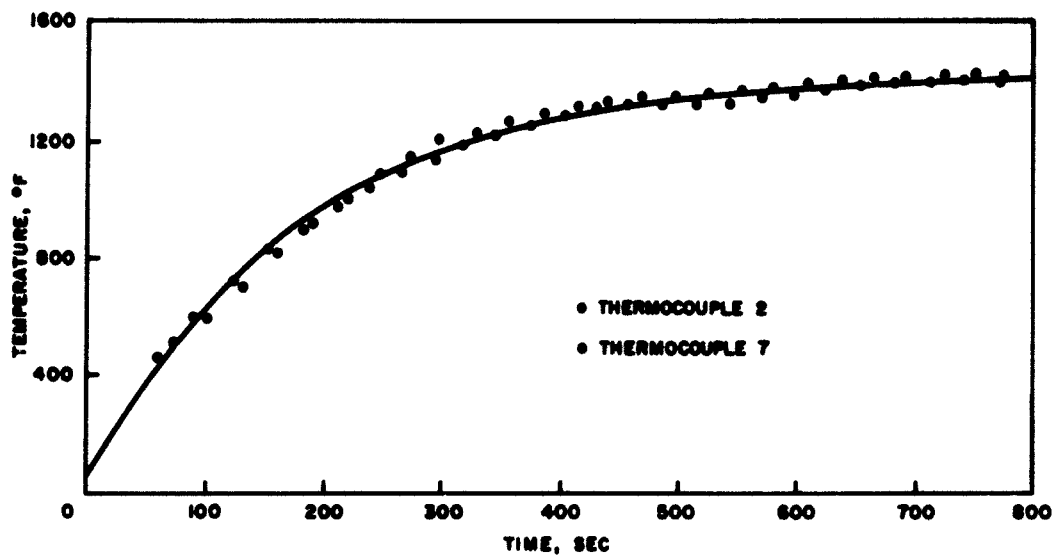


FIG. 11. Fit of Temperature-Time Curves for Silicone-Asbestos Based on Mathematical Model. Solid curve is fitted only to data from thermocouple 7.

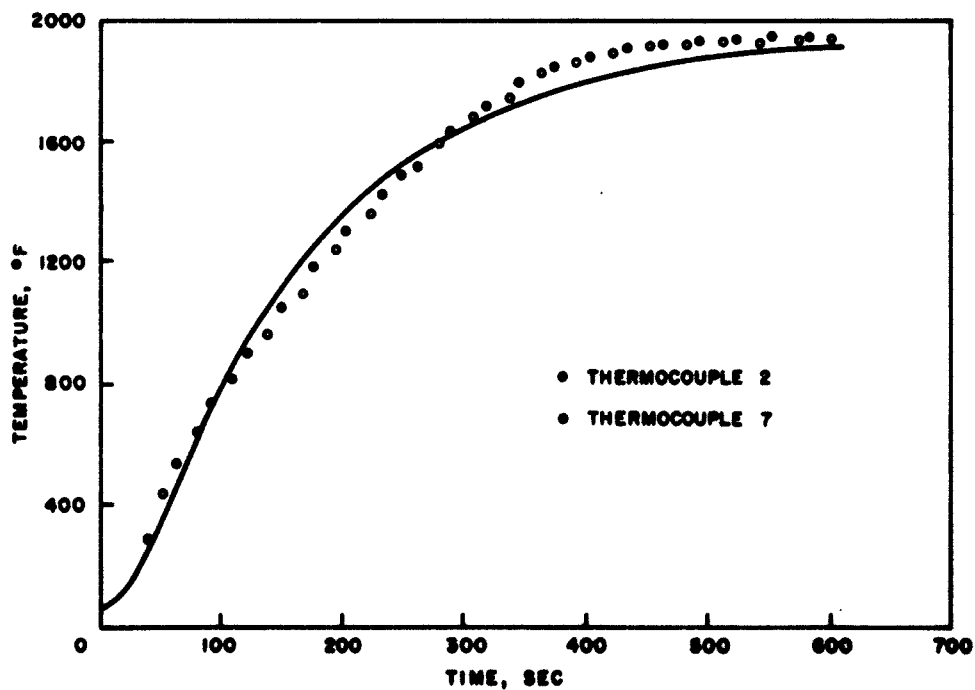


FIG. 12. Fit of Temperature-Time Curves for Phenolic-Graphite Mat Based on Mathematical Model. Solid curve is fitted to data from thermocouples 2 and 7.

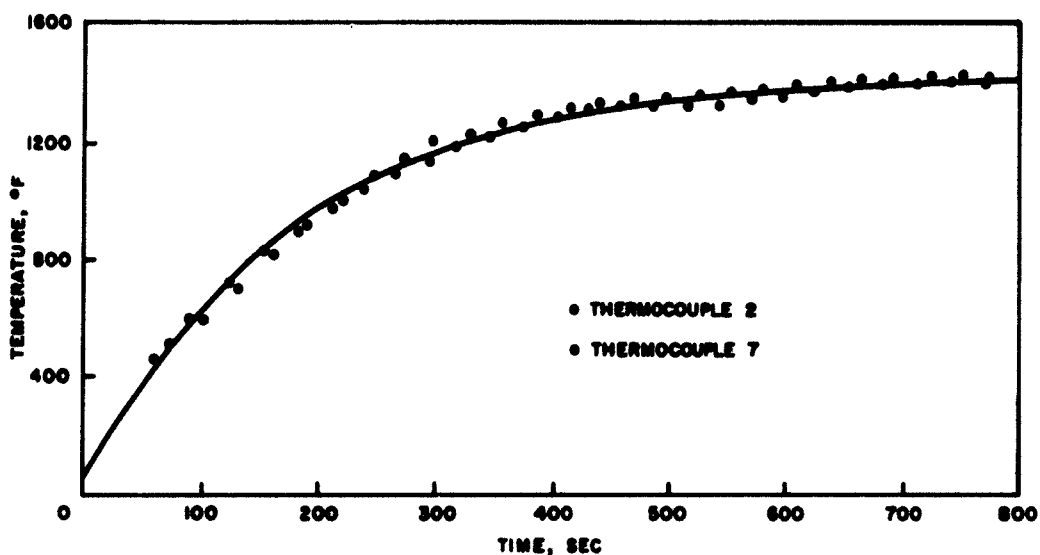


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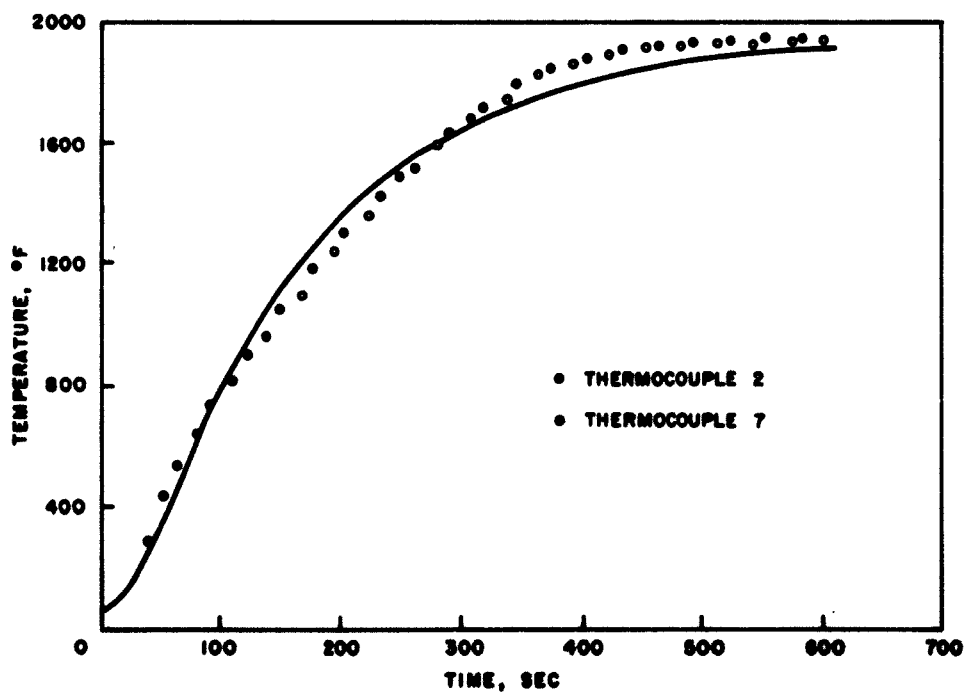


FIG. 12. Fit of Temperature-Time Curves for Phenolic-Graphite Mat Based on Mathematical Model. Solid curve is fitted to data from thermocouples 2 and 7.

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NEGATIVE NUMBERS OF ILLUSTRATIONS

Fig. 1, LHL L067706	Fig. 4, LHL L067414
Fig. 2, LHL L067707	Fig. 5-12, none
Fig. 3, LHL L067412	

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ABSTRACT. This report discusses a study conducted at the Naval Ordnance Test Station (NOTS) on the thermal properties of reinforced-plastic laminates at high temperatures. Thermal diffusivity data are given for five different laminates, and the ablation rate in mils per second is given for 15 different laminates.



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